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Supplementary material

SUPPLEMENTARY MATERIAL TO

Design, synthesis and antibacterial activity of new phthalazinedione derivatives

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Analytical and spectral data of the synthesized compounds

Compound ***3a***. Yellow crystals; yield: 65 %, 2.89 g; m.p. 330 °C. Calcd. for $C_{28}H_{19}N_3O_3$ (445.47): C, 75.49, H; 4.30; N, 9.43 %. Found: C, 75.57; H, 4.38; N, 9.50 %. IR (KBr, cm^{-1}): 3345 (NH), 2856 (aliphatic C–H), 2214 (CN), 1718 (2CO), 1662 (CO). ^{1}H-NMR (300 MHz, $CDCl_3$, δ / ppm): 3.2 (2H, *s*, C_{11}–H, C_{12}–H), 4.7 (2H, *s*, C_9–H, C_{10}–H), 7.5–7.7 (13H, *m*, Ar-H), 7.8 (1H, *s*, C=CH–Ar), 10.5 (1H, *s*, NH). ^{13}C-NMR (75 MHz, $CDCl_3$, δ / ppm): 173.8, 163.6, 145.1, 142.3, 139.4, 133.3, 129.9, 129.0, 128.5, 126.1, 125.8, 124.5, 123.7, 118.3, 112.4, 44.6, 44.4. MS (*m*/*z*, (relative abundance, %)): 445 (M^+, 0.6), 378 (0.15), 347 (0.1), 275 (5.3), 204 (0.9), 202 (3.5), 178 (100), 101 (3.5), 89 (11.5), 76 (6.2), 44 (2.0).

Compound ***3b***. Pale yellow powder; yield: 60 %, 2.85 g; m.p. 324 °C. Calcd. for $C_{29}H_{21}N_3O_4$ (475.49): C, 73.25; H, 4.45; N, 8.84 %. Found: C, 73.20; H, 4.39; N, 8.97 %. IR (KBr, cm^{-1}): 3330 (NH), 2863 (aliphatic C–H), 2220 (CN), 1721 (2CO), 1658 (CO). ^{1}H-NMR (300 MHz, $CDCl_3$, δ / ppm): 3.2 (2H, *s*, C_{11}–H, C_{12}–H), 3.8 (*s*, 3H, OCH_3), 4.7 (2H, *s*, C_9–H, C_{10}–H), 7.0–7.5 (12H, *m*, Ar–H), 7.7(1H, *s*, C=CH–Ar), 10.5 (1H, *s*, NH). ^{13}C-NMR (75 MHz, $CDCl_3$ δ / ppm): 174.0, 163.8, 160.8, 144.6, 142.2, 139.2, 131.1, 126.3, 126.0, 123.2, 124.7, 124.0, 119.1, 114.3, 109.7, 55.3, 44.6, 44.5.

Compound ***4a***. Yield: 61 %, 1.01 g; m.p. 303 °C. Calcd. for $C_{33}H_{31}N_3O_3S$ (549.68): C, 72.11; H, 5.68; N, 7.64 %. Found: C, 72.28; H, 5.74; N, 7.76 %. IR (KBr, cm^{-1}): 3270 (NH), 2939 (aliphatic C–H), 1718 (2CO), 1652 (CO). ^{1}H-NMR (300 MHz, $CDCl_3$, δ / ppm): 1.4–2.9 (19H, *m*, $9CH_2$, NH), 3.2–3.3 (2H, *s*, C_{11}–H, C_{12}–H), 4.8 (2H, *s*, C_9–H, C_{10}–H), 7.1–7.8 (8H, *m*, Ar-H). ^{13}C-NMR (75 MHz, $CDCl_3$, δ / ppm): 194.8, 174.6, 173.7, 141.3, 139.5, 138.3, 128.2, 127.1, 126.9,

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126.6, 125.2, 125.0, 124.2, 78.5, 45.3, 45.0, 41.8, 38.3, 32.0, 26.9, 25.7, 25.3, 24.9, 24.5, 23.8, 23.1, 22.1, 21.8. MS (*m*/*z*, (relative abundance, %)): 549 (M^+, 27.0), 506 (9.8), 493 (3.5), 451 (0.1), 371 (7.9), 328 (8.8), 275 (1.7), 259 (9.7), 193 (5.3), 178 (100), 151 (26.5), 123 (2.6), 78 (15.0), 44 (6.6).

Compound ***4b***. Yield: 67 %, 1.05 g; m.p. 274 °C. Calcd. for $C_{31}H_{27}N_3O_3S$ (521.63): C, 71.38; H, 5.22; N, 8.06 %. Found: C, 71.45; H, 5.31; N, 8.17 %. IR (KBr, cm^{-1}): 3266 (NH), 2945 (aliphatic C–H), 1725 (2CO), 1660 (CO). ^{1}H-NMR (300 MHz, $CDCl_3$, δ / ppm): 1.4–3.0 (15H, *m*, $7CH_2$, NH), 3.4 (2H, *s*, C_{11}–H, C_{12}–H), 4.9 (2H, *s*, C_9–H, C_{10}–H), 7.1–7.7 (8H, *m*, Ar–H).

Compound ***5a***. Yield: 75 %, 3.07 g; m.p. 306 °C. Calcd. for $C_{25}H_{18}N_2O_4$ (410.42): C, 73.16; H, 4.42; N, 6.83 %. Found: C, 73.21; H, 4.53; N, 6.92 %. IR (KBr, cm^{-1}): 3387 (OH), 3260 (NH), 1724 (2CO), 1659 (CO). ^{1}H-NMR (300 MHz, DMSO-d_6, δ / ppm): 3.2 (2H, *s*, C_{11}–H and C_{12}–H), 4.9 (2H, *s*, C_9–H and C_{10}–H), 7.0–7.8 (12H, *m*, Ar–H), 10.8 (1H, *s*, OH), 11.4 (1H, *s*, NH). ^{13}C-NMR (75 MHz, DMSO-d_6, δ / ppm): 177.4, 173.8, 159.1, 142.3, 139.4, 135.2, 129.4, 127.2, 126.8, 125.3, 124.7, 119.6, 117.7, 114.3, 44.9, 44.7.

Compound ***5b***. Yield: 77 %, 3.3 g; m.p. 328 °C. Calcd. for $C_{25}H_{17}ClN_2O_3$ (428.87): C, 70.01; H, 4.00; N, 6.53 %. Found: C, 70.08; H, 4.06; N, 6.61 %. IR (KBr, cm^{-1}): 3374 (NH), 2964, 2927 (aliphatic C–H), 1727 (2CO), 1661 (CO). MS (*m*/*z*, (relative abundance, %)): 430 (M^++2, 2.6), 428 (M^+, 8.0), 383 (0.7), 319 (2.7), 277 (1.8), 253 (1.1), 204 (1.2), 202 (6.2), 178 (100), 139 (40.7), 105 (17.6), 77 (8.0), 55 (1.7).

Compound ***5c***. Yield: 86 %, 3.4 g; m.p. 322 °C. Calcd. for $C_{24}H_{17}N_3O_3$ (395.41): C, 72.90; H, 4.33; N, 10.63 %. Found: C, 72.96; H, 4.38; N, 10.74 %. IR (KBr, cm^{-1}): 3163 (NH), 2996 (aliphatic C–H), 1729 (2CO), 1660 (CO). MS (*m*/*z*, (relative abundance, %)): 395 (M^+, 10.6), 370 (0.2), 316 (0.4), 275 (0.3), 231 (0.1), 202 (3.5), 178 (100), 152 (1.7), 106 (3.5), 78 (1.7).

Compound ***5d***. Yield: 72 %, 3.1 g; m.p. 250 °C. Calcd. for $C_{24}H_{18}N_2O_4S$ (430.48): C, 66.96; H, 4.21; N, 6.51 %. Found: C, 67.04; H, 4.33; N, 6.68 %. IR (KBr, cm^{-1}): 3166 (NH), 2959 (aliphatic C–H), 1718, 1662 (2CO), 1357 (SO_2N). ^{1}H-NMR (200 MHz, DMSO-d_6, δ / ppm): 3.1 (2H, *s*, C_{11}–H, C_{12}–H), 4.8 (2H, *s*, C_9–H, C_{10}–H), 7.1–7.8 (13H, *m*, Ar-H), 10.8 (1H, *s*, NH).

Compound ***6***. Yield: 93 %, 0.75 g; m.p. 282 °C. Calcd. for $C_{26}H_{20}N_2O_5S$ (472.51): C, 66.09; H, 4.27; N, 5.93 %. Found: C, 66.12; H, 4.30; N, 5.98 %. IR (KBr, cm^{-1}): 2880 (aliphatic C–H), 1707, 1673 (2CO), 1380 (SO_2N).

Compound ***7***. Yield: 82 %, 1.4 g; m.p. 269 °C. Calcd. for $C_{31}H_{24}N_2O_6S_2$ (584.66): C, 63.68; H, 4.14; N, 4.79 %. Found: C, 63.76; H, 4.26; N, 4.89 %. IR (KBr, cm^{-1}): 2910 (aliphatic C–H), 1732 (CO), 1387 (SO_2N). ^{1}H-NMR (200 MHz, DMSO-d_6, δ / ppm): 2.4 (3H, *s*, CH_3), 3.2 (2H, *s*, C_{11}–H, C_{12}–H), 4.7 (2H, *s*, C_9–H, C_{10}–H), 6.8–7.6 (17H, *m*, Ar–H).

Compound ***8a***. Yield: 80 %, 3.48 g; m.p. 257 °C. Calcd. for $C_{26}H_{21}N_3O_3$ (423.46): C, 73.74; H, 5.00; N, 9.92 %. Found: C, 73.64; H, 4.92; N, 9.85 %. IR (KBr, cm^{-1}): 3369, 3200 (2NH), 1727 (2CO), 1660 (CO).

Compound ***8b***. Yield: 62 %, 2.71 g; m.p. 248 °C. Calcd. for $C_{27}H_{23}N_3O_3$ (437.49): C, 74.12; H, 5.30; N, 9.60 %. Found: C, 74.23; H, 5.43; N, 9.74 %. IR (KBr, cm^{-1}): 3386, 3197 (NH), 2939 (aliphatic C–H), 1717 (2CO), 1658 cm^{-1} (CO). ^{1}H-NMR (200 MHz, DMSO-d_6, δ / ppm): 2.4 (3H, *s*, CH_3), 3.2 (2H, *s*, C_{11}–H, C_{12}–H), 4.7 (2H, *s*, C_9–H, C_{10}–H), 4.8 (1H, *s*, NH), 5.4 (2H, *s*, CH_2), 6.8–7.4 (12H, *m*, Ar–H), 9.4 (1H, *s*, NH). MS (*m*/*z*, (relative abundance, %)): 437 (M^+, 3.2), 259 (1.1), 202 (11.3), 178 (100), 120 (9.5), 91 (33).

Compound ***8c***. Yield: 75 %, 3.4 g; m.p. 260 °C. Calcd. for $C_{26}H_{20}ClN_3O_3$ (457.91): C, 68.20; H, 4.40; N, 9.18 %. Found: C, 68.27; H, 4.48; N, 9.27 %. IR (KBr, cm^{-1}): 3365, 3210 (2NH), 1725 (2CO), 1658 (CO).

Compound ***9a***. Yield: 78 %, 0.6 g; m.p. 274 °C. Calcd. for $C_{28}H_{22}N_2O_3$ (434.49): C, 77.40; H, 5.10; N, 6.45 %. Found: C, 77.48; H, 5.23; N, 6.53 %. IR (KBr, cm^{-1}): 2963 (aliphatic C–H), 1737 (2CO), 1732 (CO). MS (*m*/*z*, (relative abundance, %)): 435 (M^+, 14.0), 391 (0.9), 347 (2.2), 288 (0.8), 257 (5.3), 243 (2.2), 203 (7.0), 178 (100), 161 (5.3), 105 (22.6), 91 (9.7), 77 (1.3).

Compound ***9b***. Yield: 70 %, 0.52 g; m.p. 275 °C. Calcd. for $C_{29}H_{24}N_2O_3$ (448.51): C, 77.66; H, 5.39; N, 6.25 %. Found: C, 77.72; H, 5.46; N, 6.33 %. IR (KBr, cm^{-1}): 2867 (aliphatic C–H), 1727 (2CO), 1718 (CO).

Compound ***9c***. Yield: 80 %, 0.64 g; m.p. 292 °C. Calcd. for $C_{28}H_{21}ClN_2O_3$ (468.93): C, 71.72; H, 4.51; N, 5.97 %. Found: C, 71.69; H, 4.47; N, 5.95 %. IR (KBr, cm^{-1}): 2851 (aliphatic C–H), 1742 (2CO), 1730 (CO). ^{1}H-NMR (200 MHz, DMSO-d_6, δ / ppm): 3.2 (2H, *s*, C_{11}–H, C_{12}–H), 4.7 (2H, *s*, C_9–H, C_{10}–H), 5.4 (2H, *s*, NCH_2CO), 6.2 (2H, *s*, NCH_2N), 6.8–7.6 (12H, *m*, Ar–H). MS (*m*/*z*, (relative abundance, %)): 471 (M^++2, 0.12), 469 (M^+, 0.4), 291 (0.1), 178 (100), 138 (18.8), 75 (7.1).

Compound ***10***. Yield: 70 %, 1.22 g; m.p. 267–268 °C (glacial acetic); yield: 80 %, 1.4 g; m.p. 269 °C (THF). Calcd. for $C_{19}H_{16}N_4O_2S$ (364.42): C, 62.62; H, 4.43; N, 15.37 %. Found: C, 62.70; H, 4.46; N, 15.42 %. IR (KBr, cm^{-1}): 3409, 3248, 3142 (NH, NH_2), 1775, 1736 (2CO), 1461, 1111 (CSNH). MS (*m*/*z*, (relative abundance, %)): 348 (M^+, 1.0), 275 (0.7), 202 (3.2), 178 (100), 101 (9.7), 84 (6.4).

Compound ***11***. Yield: 84.7 %, 0.503 g; m.p. 197 °C. Calcd. for $C_{19}H_{15}N_3O_2S$ (349.41): C, 65.31; H, 4.33; N, 12.03 %. Found: C, 65.38; H, 4.39; N, 12.07 %. IR (KBr, cm^{-1}): 3136, 3111 (2NH), 2937–2866 (OH), 1709 (CO), 1461, 1256 (CSNH). MS (*m*/*z*, (relative abundance, %)): 203 (ethenoanthracene, 3.0), 178 (100), 152 (13.0), 81 (11.7), 59 (13.0).

Compound ***12***. Yield: 85 %, 0.7 g; m.p. 229 °C. Calcd. for $C_{24}H_{25}N_3O_2S$ (419.54): C, 68.71; H, 6.01; N, 10.02 %. Found: C, 68.80; H, 6.07; N, 10.06 %.

IR (KBr, cm^{-1}): 3172 (NH), 2949–2851 (OH), 1703 (CO). ^{1}H-NMR (200 MHz, DMSO-d_6, δ / ppm): 0.8 (3H, *t*, CH_3), 1.2–1.7 (6H, *m*, $3CH_2$), 3.2 (2H, *br*, *s*, C_{11}–H, C_{12}–H), 3.6 (2H, *t*, SCH_2), 4.7 (2H, *br*, *s*, C_9–H, C_{10}–H), 7.1–7.3 (8H, *m*, Ar-H), 11.5 (1H, *s*, NH), 12.3 (1H, *s*, OH). MS (*m*/*z*, (relative abundance, %)): 420 (M^+, 0.6), 375 (1.1), 347 (0.5), 330 (0.6), 241 (1.3), 197 (1.4), 194 (4.7), 178 (100), 97 (3.3), 51 (3.0).